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Abstract

A low-cost chemical sensor comprising surface-mount light-emitting diodes (LEDs) has been developed for colorimetric gas detection. The device consists of a pair of LEDs connected to a simple PIC microcontroller circuit and in the most basic form, requires the use of only two input-output (I/O) pins on the chip. The key features of this sensor are the use of a LED rather than a photodiode for light detection and an all-digital light detection protocol that leads to a reduction in cost and power consumption by avoiding the need for an analog-to-digital converter. The surface-mount diodes employed are more compact than standard LEDs and are more amenable to coating by solid-state sensor films. Results from sensors employing a chemochromic ammonia sensitive film are presented, and the detection of this target is demonstrated in the parts-per-million range. The configuration is applicable to a wide range of colorimetric gas sensing materials.

Keywords

led, mount, gas, surface, low, sensor, cost

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Low-Cost Surface-Mount LED Gas Sensor

Roderick L. Shepherd, William S. Yerazunis, *Senior Member, IEEE*, King Tong Lau, and Dermot Diamond

***Abstract*—A low-cost chemical sensor comprising surface-mount light-emitting diodes (LEDs) has been developed for colorimetric gas detection. The device consists of a pair of LEDs connected to a simple PIC microcontroller circuit and in the most basic form, requires the use of only two input–output (I/O) pins on the chip. The key features of this sensor are the use of a LED rather than a photodiode for light detection and an all-digital light detection protocol that leads to a reduction in cost and power consumption by avoiding the need for an analog-to-digital converter. The surface-mount diodes employed are more compact than standard LEDs and are more amenable to coating by solid-state sensor films. Results from sensors employing a chemochromic ammonia sensitive film are presented, and the detection of this target is demonstrated in the parts-per-million range. The configuration is applicable to a wide range of colorimetric gas sensing materials.**

***Index Terms*—Ammonia sensor, colorimetry, gas sensor, light-emitting diodes (LEDs), LED sensor, low-cost sensor, optical sensor.**

I. Introduction

LIGHT-EMITTING diodes (LEDs) are being used far more commonly as light sources in optical chemical sensors due to the low-cost, low-power consumption, reliability, and ever-increasing range of devices and wavelengths available. The increased interest in LED sources has had a major impact on low-cost-component-based chemical sensors, where the main goal is to achieve analytical performance without the expense of more conventional instrumentation [1]–[5]. Typically, a photodiode is used for detection, good sensitivity, and a significant reduction in system cost. Usually, the photodiode is operated at $V_{\rm bias} = 0$ V and hence can be considered as a low-power sensor. However, in addition to the detector, a good-quality operational amplifier and mid-to-high-resolution analog-to-digital (ADC) are required to complete the device. These additional components not only increase system complexity and cost but also add to the power requirements, which is of particular importance in battery-powered systems. The sensor we have developed requires only a simple microcontroller and a pair of LEDs, one of which is driven in reverse-bias mode to function as a light detector. The detector LED requires a short reverse-biasing step (100 μs) to the supply voltage (3 or 5 V), but there are no other components requiring additional power, so this is the only power draw associated with the detector itself. The system provides a simple low-cost battery-powered sensing platform with very good sensitivity and signal-to-noise (S/N) characteristics. Initially, we developed this system for communication and light sensing applications that made use of the bidirection properties of LEDs [6]; after which, we began work on all-LED colorimetric chemical sensors. To date, we have reported on the use of a fused-LED sensor for monitoring colorimetric reactions in solution [7] and on the detection of Fe(II) at concentrations in the low-parts-per-billion (ppb) range using a pair of LEDs in transmittance mode [8]. Recently, we presented results on a transmission-mode solid-state gas sensor for ammonia, which comprises two small surface-mount LEDs and a chemochromic polymer sensing film [9]. This paper provides a more detailed account of the surface-mount LED ammonia sensor and outlines the key advantages of using this approach for colorimetric chemical sensing.

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II. Materials and Methods

A. Sensing Protocol

The sensing technique that we have developed is all-digital in nature; hence, there is no need for conversion of an analog signal, which is normally the case with photodiode-based optical chemical sensors. This reduces system cost by avoiding the need for a precision operational amplifier (OPAMP) and an external ADC chip or a more expensive microcontroller with a reasonably high-resolution built-in ADC. There is a short reverse-biasing step (100 μs) associated with our LED sensor, which involves charging the diode capacitance to the supply voltage (3 or 5 V), but this is the only power draw involved. While photodiode systems are typically operated at $V_{\rm bias} = 0$ V, the power consumption of any supporting chips required for this method of detection (OPAMP and usually external ADC) will be significant. Hence, the LED-based sensing approach is low cost with relatively low power requirements, and our findings thus far indicate that the signal integration approach used in the digitization of the photocurrent gives excellent S/N characteristics and therefore, very low limit of detection (LOD) in analytical measurements [8].

Typically, the detector LED is connected to a pair of digital input–output (I/O) pins and operated in reverse-bias mode. However, if pin availability is critical, the detector can be driven from a single I/O pin with the anode tied to ground. The advantage of using a pair of I/O pins for detection is that the LED can be operated bidirectionally and hence can be used to emit and detect light. This has potential implications for sensors with communication capabilities, ranging from simple visual feedback to more complex line-of-sight data transfer. In either case,

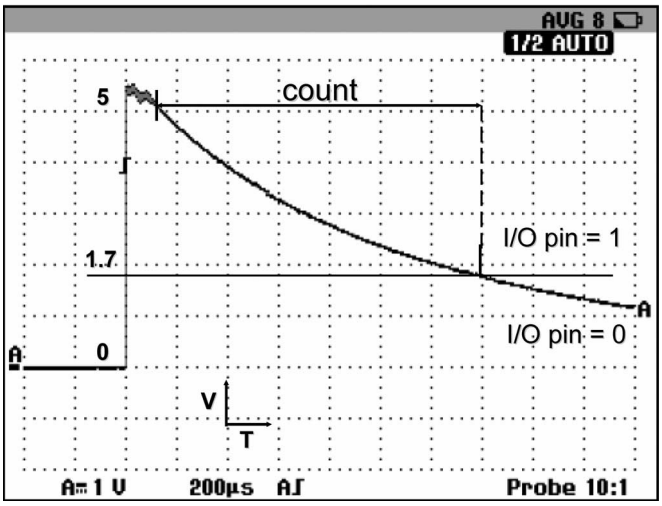

Fig. 1. Oscilloscope trace of the LED sensor discharge curve. Digital detection of light is achieved by continually polling the I/O pin logic state.

the diode junction is charged to the supply voltage (normally 5 V), and then the I/O pin connected to the cathode is switched to high-impedance input mode. Photons impinging on the detector diode produce a small (in nanoamperes) photocurrent, which causes the 5 V charge to decay. Using a timing routine implemented in firmware that is programmed onto the microcontroller, we continually poll the logic state of the input pin until the logic 0 threshold ($\sim$ 1.7 V) is reached. The time taken in microseconds to reach logic 0 is proportional to the amount of light detected. As the amount of light received increases, the diode discharges more rapidly, and hence, lower timing values are observed. Conversely, as the amount of light decreases, the time to reach logic 0 increases. An oscilloscope trace of a sensing cycle (Fig. 1) illustrates this process of diode discharge and logic-threshold crossing. The logic voltage of the I/O pin is plotted on the vertical axis, whereas time is plotted along the horizontal axis in 200 μs divisions. The time-based measurement in this example is indicated by the horizontal line labeled "count" and corresponds to a value of approximately 1300 μs. To increase noise immunity, the measurement process is not a simple timer that stops when the first logic 0 is read. Instead, repeated measurements are performed in rapid succession over the maximum time interval we wish to accept. For each measurement showing a logic 1, we increment a counter; if the measurement shows a logic 0, we do not change the counter. Assuming that the noise signal is uncorrelated with the timing of the sample measurements, this gives us a significant improvement in the S/N ratio and also yields a constant and deterministic output rate for the sensor–microcontroller system. At present, data is collected via an RS232 link between the device and a PC, but this could be easily replaced with a LED-based wireless communication link.

The circuit employed for this paper (Fig. 2) comprised a PIC16F876 microcontroller, MAX232ACP RS232 level shifter, and supporting components required by the chips, including a 20 MHz ceramic resonator for the PIC. The microcontroller has a built-in 10-bit ADC, but this feature is not required for our sensor and hence was unused. While many reasonably priced microcontrollers, such as the PIC16F876, now have an internal

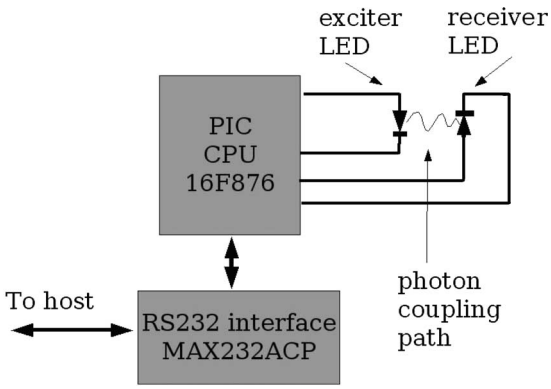

Fig. 2. Block diagram of the PIC16F876 LED sensing circuit.

ADC, these currently tend to be more of general-purpose converters, where the resolution is only 10 bits. In many sensing applications, better resolution is required, so typically a 12-bit or even 16-bit external ADC is employed, which adds significant cost to the system and increases power usage. The current PIC16F876-based circuitry provides a generic interface and control platform that is used for the development of a range of different sensors in our laboratories, but the sensing technique described herein could easily be implemented on a lower cost microcontroller without an ADC. The 16F876 is running at 5 million instructions per second (MIPS), and the resulting minimum sensing resolution achievable at this clock speed is approximately 5 μs. The maximum fixed sensing period typically employed is 65 000 μs, which ensures that a suitable integration time is provided without causing any overflow problems (timing values are stored in a 16-bit variable $\max = 65535$). Significantly longer integration periods are possible by adding an additional delay in the timing routine implemented in the software on the PIC. This scaling permits measurements to be made over hundreds of milliseconds or even seconds if required, but there is an obvious tradeoff between the total integration time and resolution, which decreases with increased scaling.

If noise is considered (typically ± 10 μs on the observed discharge time), then the minimum sensing resolution is closer to 40 μs, but this still provides more than adequate sensitivity for the measurements described in this paper.

B. Sensor Design

The sensor comprises two 1206-format surface-mount LEDs, which have overall dimensions of 3.2 $\times$ 1.6 mm and a flat lens (Fig. 3). For the sensor described in this paper, a 574 nm green exciter LED (Kingbright KP-3216MGC) was used in combination with a 621 nm red-orange detector LED (Lite-On LTST-C150KAKT). The LEDs are soldered onto 2.0 mm Molex pitch header pins for ease of connection and to allow for adjustment of the air gap (Figs. 3(b) and 4). A variety of arrangements are possible, but for this paper, we focused on the design shown in Fig. 4, where the diodes are stacked vertically with respect to each other. For this paper, the detector LED, which is coated with the chemochromic reagent film, was positioned such that it faced downward, effectively reducing effects arising from variations in the ambient light. In particular,

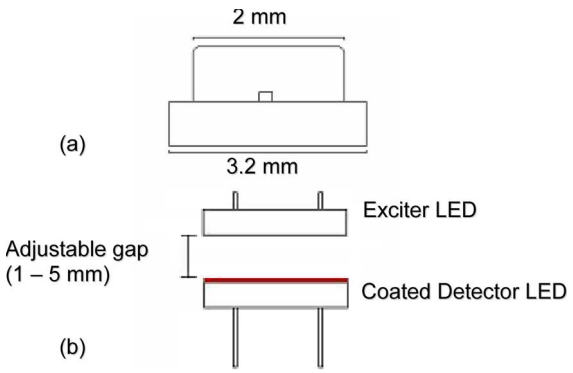

Fig. 3. (a) Schematic of the surface-mount LED ammonia sensor illustrating the form factor of the 1206 surface-mount LEDs and (b) configuration of LEDs in the chemical sensor. (Color version available online at http://ieeexplore.ieee.org.)

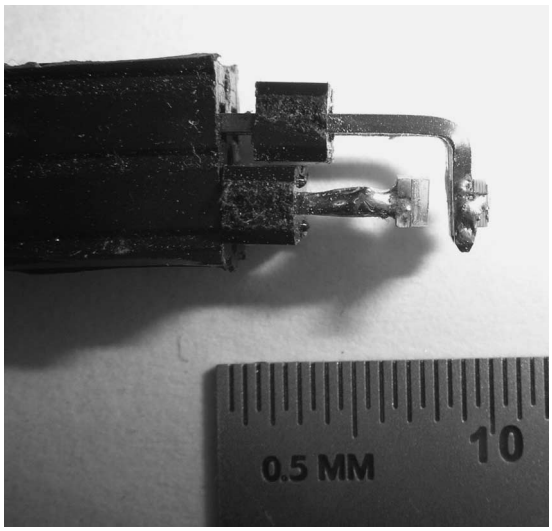

Fig. 4. Surface-mount LED chemical sensor housed in a Molex receptacle. Coarse baseline adjustment is made by changing the air gap between the LEDs.

this arrangement ensures that the majority of room lighting does not impinge directly on the detector diode. The exciter LED, which was used without any additional coating on the lens, was positioned directly below the detector at a distance of approximately 0.5–1.0 mm. The LEDs were plugged into a 2.0 mm Molex receptacle, and the air gap between the LEDs was adjusted by simply moving the detector LED relative to the exciter (Fig. 4). This physical adjustment of the LEDs with respect to each other allows for coarse tuning of the baseline signal, which is particularly useful when the relocation of a device involves major changes in ambient light levels.

C. Ammonia-Specific Colorimetric Reagent

The reagent used for this paper is p-nitrophenylnitrosamine (NPNA) and was synthesized in-house (Fig. 5). A mix of NPNA and polyvinyl chloride (PVC) in cyclohexanone was prepared, which was subsequently drop coated onto the detector LED lens by applying a small volume from a pipette. Due to the relatively low surface area of the LEDs used in this paper, the volume of the reagent required for preparation of each sensor was kept to a minimum ($< 5\ \mu$L). NPNA forms a weak complex with

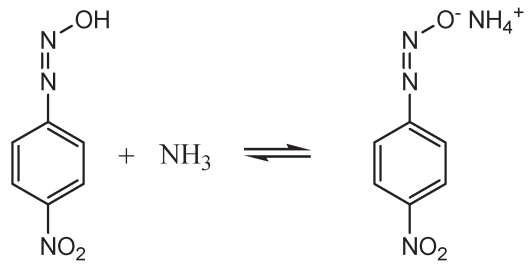

Fig. 5. Reaction scheme of NPNA with NH_3. Ammonia forms a weak complex with NPNA, which brings about a color change from yellow to red.

ammonia (Fig. 5) and causes a chromic shift in the material from yellow to red. NPNA is pH sensitive to some extent, but only extremely basic species shift the equilibrium. As there are limited cross-reacting species in the gas phase with these properties, NPNA in the solid state exhibits reasonably high selectivity to NH_3 and primary amines.

D. Ammonia Sensor Testing

The gas sensor was tested for responses to NH_3 in a simple flow cell that was fabricated in-house. A 100 ppm (v/v) NH_3 standard premixed gas cylinder (Scott U.K.) was used in combination with a nitrogen line for dilution to deliver ammonia samples in the concentration range of 6.25–14.3 ppm to the sensor. Gas flow rates were controlled by rotameters and were in the range of 0–50 mL/min for NH_3 and constant 300 mL/min for N_2. The nitrogen flow was maintained at 300 mL/min throughout all experiments, both during the response and recovery cycles, as the manual adjustment on the rotameters used was not precise enough to allow for continual flow adjustments. The gas flow was passed through the headspace of a water bubbler to maintain relatively constant humidity throughout the experiments.

III. Results and Discussion

A. NPNA Optical Response

The optical response of NPNA upon exposure to ammonia is due to shifts of the protonated-form absorbance peak ($\lambda \max = 420$ nm) to the deprotonated basic form ($\lambda \max = 550$ nm) (Fig. 6). This causes a visible color change in the material, which is yellow in air and red after interaction with ammonia. The green emitter LED chosen for this particular sensor has a peak wavelength of 574 nm, a dominant wavelength of 568 nm, and a spectral half-width of approximately 20 nm [10]. The emission spectrum of the LED was measured to confirm these specifications using an Ocean Optics S2000 spectrometer and has been superimposed over the NPNA spectra in Fig. 6 for comparative purposes. It is clear that the emissive properties of this LED overlap well with the base form of NPNA, and hence make it an ideal light source for monitoring the development of the red NPNA/NH_3 complex. As the concentration of NH_3 increases, the intensity of light transmitted through the sensing film to the detector LED decreases, and the discharge time values measured by the microcontroller circuit increase.

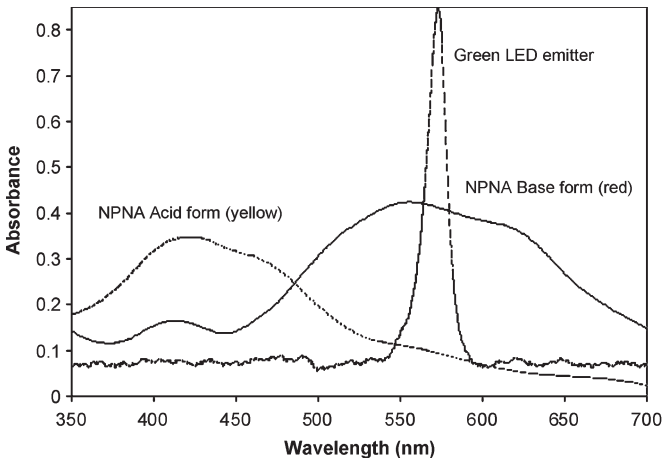

Fig. 6. Spectra of the NPNA acid and base forms, and emission spectrum of a 574 nm green emitter LED. The overlap between the LED source and base form of NPNA, which develops in the presence of ammonia, is very good.

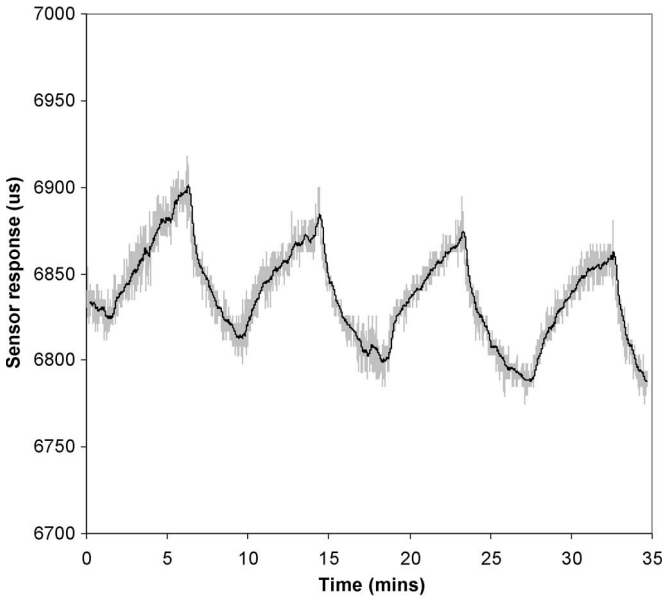

Fig. 7. Response of the LED sensor to multiple samples of 9 ppm NH_3 in nitrogen. Raw data is plotted in gray, and the smoothed response is plotted in black after a moving average filter (per $= 20$) was applied to the raw data. Increases in the sensor timing response are observed in the presence of ammonia due to stronger absorbance of emitted light by the NPNA film.

B. LED Sensor Responses

The sensor was exposed to NH_3 samples in the range of 6.25–14.3 ppm. As mentioned earlier, an approximate lower sensing limit of this device based upon observations of the baseline noise is around 20 μs of dither in the measured nominal discharge time. Thus, at lower concentrations of the NH_3 tested, the sensor was approaching the LOD. Sensor responses to multiple samples of 9 ppm NH_3 are shown in Fig. 7, and as expected, increases in the sensor timing response were observed in the presence of ammonia. The level of noise present in the signal is quite significant, but despite this, the responses are clearly distinguishable and reproducible. By applying a simple moving average filter to data (Microsoft Excel, period $= 20$), there is a significant improvement in the S/N ratio [Fig. 7, (solid line)]. Some baseline drift was observed (Fig. 7), which can

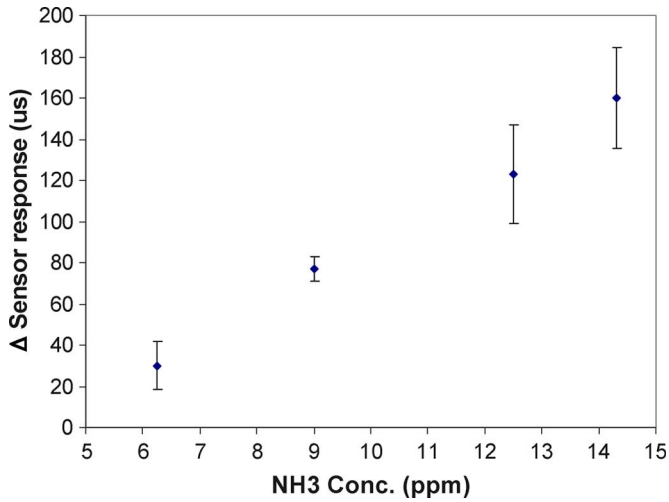

Fig. 8. Average changes in the response $(n = 4)$ of the LED ammonia sensor, with error bars indicating the standard deviation. (Color version available online at http://ieeexplore.ieee.org.)

be attributed to the equilibration of relative humidity (RH) in the sensor test chamber over the course of the experiment. The drift is most evident at the start of the test run (peak 1), with the baseline virtually stabilizing by the last sample (peak 4). Experiments were carried out at essentially saturated RH, which provided a simple means of maintaining humidity close to constant but caused some drift due to polymer swelling as the RH stabilized. Despite some baseline instability, sensor responses were not significantly affected, and quite reasonable reproducibility was observed from one sample to the next.

The simple flow system used to prepare and deliver these gas samples did not allow precise control of the NH_3 flow rates, and the influence of this on sensor reproducibility became more evident at faster rates, where the ammonia accounted for a larger fraction of the total flow. The larger errors associated with the 12.5 and 14.3 ppm data points (Fig. 8) are indicative of this flow-induced variation in the response. Despite these sources of variation, responses were linear over the entire concentration range investigated, as shown in Fig. 8. The NH_3 range studied was quite limited, as we were primarily concerned with determining or at least estimating the LOD. The minimum NH_3 concentration that could be generated with the setup used was 6.25 ppm; hence, it was not possible to obtain data at lower concentrations.

The estimated LOD, which is based on the baseline noise of the raw data and an average response at 6.25 ppm NH_3 of approximately 30 μs, is about 5 ppm in this particular configuration and experimental design. We are confident that this can be significantly improved by applying simple filtering as previously discussed and by varying the polymer sensing layer thickness, which has a significant effect on both response time and sensitivity. Until now, we have coated the detector LED in our sensors by simply drop coating a small volume of NPNA/polymer onto the lens and allowing this to dry. While this has been adequate for initial prototype sensors, the ideal approach to controlling the film thickness is spin coating. The surface-mount LEDs used are ideal for use with this deposition technique, as the lens structure provides a uniformly flat surface.

Controlling the volume of the sensing material and rotation rate will provide a means of varying the NPNA film thickness, enabling further optimization of the sensors. We plan to investigate this further in the future and are already encouraged by the results obtained using the sensor described in this paper. The current surface-mount LED sensor, which is clearly not optimized, detected ammonia in a linear fashion in the 5–15 ppm range.

C. System Flexibility and Portability

One of the key features of our system is the software-based timing protocol. In addition to the aforementioned benefits of digital signals this provides, many sensing parameters can be controlled and updated in the software. Critical sensing properties including integration period, minimum timing resolution, average rate, sampling rate, and power saving schemes can easily be controlled at run-time in code or updated by an external device without any change to the physical characteristics of the sensor itself. If the detector LED is configured to operate bidirectionally, these sensor properties can even be updated using LED-to-LED wireless communication.

The sensors are also highly portable, again due to the software-based technique used to measure the detector photocurrent, and are therefore effectively platform independent. LED-based sensors can be interfaced seamlessly with a wide range of different microcontroller architectures and microcontroller-based devices. The main task when moving between different processors involves porting a small piece of firmware, and this could be streamlined by developing a library of drivers to support the more common parts. Potentially, LED-based sensors can be incorporated into a diverse range of devices, but one area of major significance is the emerging field of wireless sensor networks (WSNs). These systems are battery powered microcontroller-based communication and sensing nodes that rely on low-power components and highly efficient power management in order to extend lifetime. Various platforms that are based on a range of different microcontrollers are being developed [11]–[14]. In all cases, there are versions of these platforms that run on 3 V lithium coin cells, which have small energy capacities (typically 200–600 mAh at 3 V). Power efficiency is therefore paramount, and saving a few milliamperes of current can significantly increase the lifetime of an individual node in the network. These LED-based sensors are ideally suited for hardware platforms such as these with limited power, and we are currently investigating the integration of our sensors with low-power wireless nodes. The ultimate goal is to deploy low-power WSNs for environmental and industrial monitoring, with the sensing tasks performed by low-cost LED chemical sensors.

IV. Conclusion

A low-cost colorimetric ammonia gas sensor that uses small surface-mount LEDs as both light source and detector has been developed. Sensor responses are monitored using a simple microcontroller circuit, which is entirely digital in nature. This configuration avoids the cost and power requirements of typical supporting components employed when using a photodiode (a precision OPAMP and an expensive external ADC or a more expensive microcontroller with high-resolution internal ADC), and as the sensing protocol employs a software-based photocurrent measurement, the devices are highly flexible and essentially platform independent. The sensor has been demonstrated for the detection of low-parts-per-million levels of ammonia, as determined using a selective colorimetric polymer coating that contains NPNA, but this configuration is amenable to a wide range of colorimetric-based gas sensing materials. Indeed, virtually any colorimetric assay could be immobilized in some way and deposited onto the lens of a surface-mount LED. The challenge is really in developing reliable sensing materials for this purpose, with the LED sensor providing a low-cost generic platform upon which sensors can be developed. The portability, low cost, and low-power characteristics of these devices make them ideal chemical sensors for WSN applications, where power consumption is critical.

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King Tong Lau received the Ph.D. degree in electrochemical biosensor development from University of London, London, U.K., in 2001.

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He is currently the Principal Investigator of a recently awarded Science Foundation Ireland (SFI) grant and the Director of the Adaptive Sensors Group, National Centre for Sensor Research, Dublin City University (DCU). The Adaptive Information Cluster (AIC) is an SFI-funded initiative that brings together DCU and University College Dublin and whose goal is "to capture the world digitally in a comprehendible way." As such, the AIC is integrating leading-edge research in wireless sensing, content extraction from video and audio media, collaborative reasoning, and personalization. He has published over 100 peer-reviewed papers in international science journals and is a coauthor and editor of two books, which were both published by Wiley. His research interests include molecular recognition; host–guest chemistry; ligand design and synthesis; electrochemical and optical chemical sensors and biosensors; lab-on-a-chip; sensor applications in environmental, clinical, food quality and process monitoring; development of fully autonomous sensing devices; wireless sensors; and sensor networking.